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THE COMPARATIVE SENSITIVITY OF "SUPERFINE" PETN AND DEXTRINATED LEAD AZIDE

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MARCH 1976

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The sensitivity of two different lots of a special grade of PETN, designated as "superfine" PETN was compared with that of dextrinated lead azide with respect to impact, shock, short high temperature pulses, and gaseous electrostatic discharge. Results of the seven tests employed showed that both lots were clearly less sensitive than the reference dextrinated lead azide. Responses were not markedly different than those

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20. ABSTRACT (Continued)

of standard PETN (Mil-P-387). The test for initiation sensitivity (minimum priming charge) did, however, indicate that the test explosive is more sensitive than most of the secondary explosives.

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INTRODUCTION

This study was undertaken to compare the sensitivity of a special grade of PETN--designated here as "superfine" PETN[1]--to that of dextrinated lead azide with respect to impact, shock, heat, and electric discharge. This material consists of granular crystallites about 10 microns in size.

The information was required in order to determine the acceptability of this material for NASA Space Shuttle applications. In addition, a number of comparison tests were performed on other explosives of interest to NASA, for which information in the literature was lacking or sparse.

The experimental work, described later in some detail, includes provisions for comparisons of sensitivity to impact by the Bureau of Mines method (Ref 1), and by a ball-drop method used by the Explosives Division for primary explosives (Ref 2); to shock using the "sensitivity-to-initiation" test (Ref 3) and the small scale gap test devised by the Navy (Ref 4); to short pulses of heat (Ref 5); and to electrostatic discharge by the standard method used at Picatinny Arsenal (Ref 1). In addition, a supplementary electrostatic test using a parametric procedure and an apparatus employing parallel plate electrodes (Ref 6) was also used on the grounds of greater sensitivity and closer approach to voltage and capacitance values encountered in practice.

A summary of the tests performed, and a list of the test explosives together with their identification by lot number, are given in Table 1 For convenience, the Ensign-Bickford PETN is designated as "PETN (E-B)" and the DuPont material as "PETN (DuP)." The regular grade PETN, which meets Mil-P-387 specifications, is designated without a subscript.

[1]U.S. Patent 3, 754, 061 Issued to C.D. Forest, August 1971, assigned to DuPont.

Table 1

Test plan for the explosive sensitivity study

Explosive (Identification)	Impact		Shock		Heat	Electrostatic	
	Bureau of Mines (cm)	Ball Drop (inches)	Initiation Sensitivity (g LA)	SSGT (decibands)	Hot Wire (mjoules)	Picatinny Arsenal (joules)	Explosives Division (joules)
PETN (E-B) Ensign-Bickford 9-56A	X	X	X	X	X	X	X
PETN (DuP) DuPont L-71N011A-254	X	X	X	X	X	X	X
Lead Azide, dextrinated Olin-Matheson OMC 69-104	X				X	X	X
PETN Unidynamics 5-309		X		X	X		
HNS-II* Ensign-Bickford ENB-5735-02	X		X		X		
HMX (Class B) Holston SR 070402					X		
RDX (Class B) (lot number not known)					X		

*HNS (Hexanitrostilbene): This is the fine size fraction. HNS-I is the coarser fraction.

DESCRIPTION OF THE EXPLOSIVES

Two lots of superfine PETN were tested. One was supplied by Ensign-Bickford and the other by the Pompton Lakes Facility of DuPont. Both batches were shipped under alcohol-water mixture. Laboratory portions were removed from these wet batches, filtered, and washed with methyl alcohol on a Buchner funnel, and then dried for twenty-four hours in a vacuum oven at 60-65°C. All vacuum-dried materials were kept in closed conductive rubber containers until used.

Where samples had to be prepared by pressing, the pressing operation was performed in a room kept at 25°C and 50-55% RH.

EXPERIMENTAL TESTS AND RESULTS

The term "minimum" energy (or other stimulus) when used in connection with sensitivity data refers to the lowest level at which at least one fire is obtained in ten trials. Occasionally the next lowest level is given, i.e., the highest level at which some specified number of consecutive (commonly ten) no-fires is obtained. This is referred to as a "zero" level. As a matter of prudence, however, both values should be regarded only as a measure of the level below which it is not feasible to estimate initiation probabilities; one <u>must not</u> assume that initiation cannot occur at lower levels or that an explosive is "safe" if a higher level is not used.

An alternative commonly used value for comparing sensitivities is the "50% Point," i.e., the level at which probability of initiation is 50%. This value is estimated in this work by the Bruceton method, which is described in Reference 7 under "US/Impact/02." The testing is carried out in the region where both positive and negative trials occur, and the level of stimulus is changed after each trial in accordance with a definite pattern. Thus, if a given trial is negative (no fire), the stimulus is increased to the next higher step. If the given trial is positive, the level is decreased one step. The steps are equal, and usually are chosen to approximate one standard deviation.

Impact Sensitivity (Bureau of Mines Apparatus)

Experimental

In this test (Ref 1, 7) a small amount, nominally 20 mg, of the loose powder is placed on a steel anvil of Rockwell hardness C 60-65 and a striker of similar hardness is lowered onto the powder. The surfaces of both anvil and striker are smooth. A drop weight of 2 kg mass is allowed to drop by free fall onto the striker and the result is noted. Any evidence of reaction, such as sound, smoke, or light, is taken as a "fire."

Results

Results are summarized in Table 2 and are for the minimum heights at which one explosion occurred in ten or fewer trials. Tetryl has been included in this test for reasons given in the "Discussion" following.

Discussion

It is common practice, in testing for impact sensitivity, to include one or more "standard" explosives to ensure that the apparatus is functioning properly. In the test plan of Table 1, the lead azide served this purpose; however, the result was lower than is sometimes observed (e.g., see Ref 3). Tetryl was therefore run to determine whether or not the apparatus was consistent or whether the lead azide result was anomalous. Comparison of the results obtained in this work with values for the azide and tetryl from the literature show that both are less than literature values by roughly the same amount. It was therefore assumed that a valid comparison between the superfine PETN and lead azide could be made.

The average value of the two superfine PETN samples is 15.5 cm, or very nearly twice the height for the lead azide, and indicates a significantly lower sensitivity to impact than is shown by azide.

Table 2

Summary of impact sensitivity data,
Bureau of Mine Apparatus

Explosive	10% Ht (cm)
PETN (E-B)	13
PETN (DuP)	18
Lead Azide	8
HNS	15
Tetryl	20

Impact Sensitivity (Ball Drop Apparatus)

Experimental

The essential features of the ball drop assembly (Ref 2) are a small hardened steel block, a 1/2 inch diameter ball bearing (8.33 g), a grooved track to release the ball bearing at a given height, and a ring stand to hold the assembly. The block is provided with a pair of side rails, whose height above the impact block is adjusted by means of shims to 0.013 inch. In practice, about 35 mg of explosive is put in the center of the block, and the powder is leveled off to a thickness of 0.013 inch by running a straightedge or plastic rod along the side rails. The block is centered beneath the track, whose height is selected, the ball is allowed to roll off the track and fall onto the powder, and the result noted.

Results

Results are summarized in Table 3, together with other available comparative data. Note that no fires were obtained for any of the PETN samples at the maximum available height of 45 inches. In contrast, the data for several different lots of various types of lead azide (including dextrinated) during a number of surveillance and other studies were in the range of 21.3 to 24.3 inches.

Table 3

50% heights by the ball-drop method

Explosive	50% Height, inches	
	This Work	PATR 4357
PETN (E-B)	45+	
PETN (DuP)	45+	
PETN	45+	
Lead Azide		21.3-24.3

Discussion

This test shows unequivocally that the superfine PETN powder is much less sensitive to impact than is lead azide.

Initiation Sensitivity (Minimum Priming Charge)

Experimental

In this test (Ref 3) the sample consists of 0.4 g of the test explosive, which is pressed at 3,000 psi into an empty No. 6 blasting cap. The test variable is the amount of lead azide, which is pressed on top of the test explosive. The amount of lead azide is varied in increments of 0.01 g. A steel witness disc, 1 inch diameter x 1/16 inch thick, of cold rolled steel, is used as a criterion of detonation: a positive result is indicated by a hole being blown through the disc. The procedure was to repeat trials at decreasing increments of lead azide in order to determine the largest quantity that would result in ten consecutive no-fires.

The experimental setup is shown in schematic form in Figure 1.

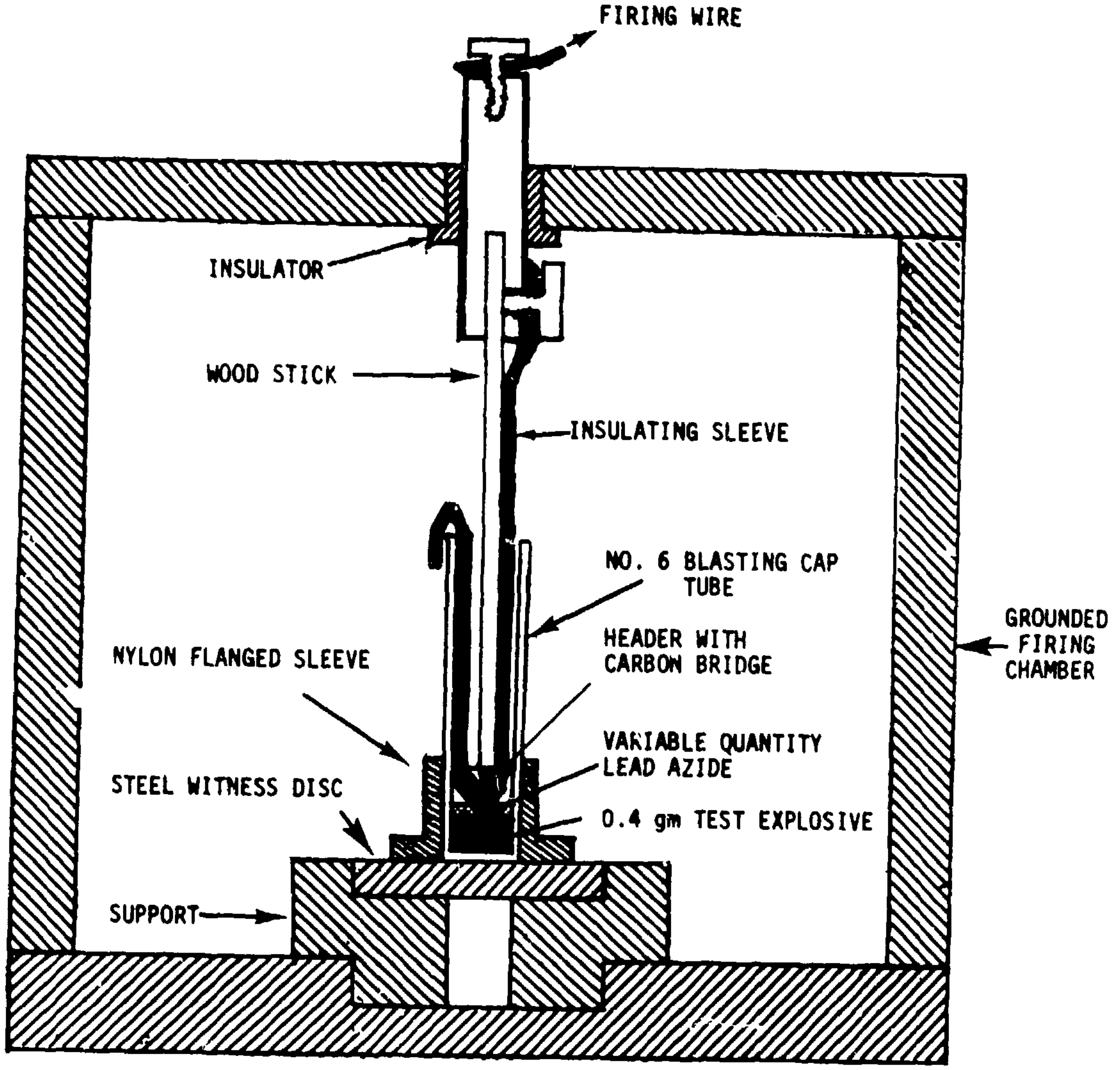

Fig 1 Schematic of minimum priming charge test

Results

The test for all PETN samples was terminated when the amount of lead azide had been reduced to 0.005 g, since it was not feasible to use lesser amounts. Results are summarized in Table 4. The procedure is also shown in Table 5. PETN samples could not be fired without lead azide.

Discussion

A direct comparison of shock sensitivity between PETN and lead azide cannot be made in this test; however, it is apparent that the superfine PETN should offer no problem in application due to difficulty in initiation.

Small-Scale Gap Test

Experimental

In this test (Ref 4) an explosive is subjected to an attenuated shock from a standard source. The source consists of a column of RDX in a 1.00-inch-OD brass tube having an ID of 0.204 inch and 1.50 inch length. The RDX is pressed to a loading density of 1.56 g/cc. The test explosive is pressed at 16,000 psi into a similar brass tube in seven increments in order to achieve as uniform a loading density as possible. Attenuation is produced by inserting various thicknesses of polymethylmethacrylate (PMMA) between the donor and the test explosive (the acceptor). A 1/2-inch-thick block is used as a witness plate to determine the occurrence of a fire. Two shots are fired without any attenuation and the depth of the dent produced in the block is measured. In subsequent shots, a dent equal to or greater than 1/2 the "barefoot" dent is taken arbitrarily as a fire. The arrangement is illustrated in Figure 2 except that the chamber used to contain the explosion is not indicated. The shock strength in "decibangs" is defined by the equation:

$$S \text{ (decibangs)} = 30 - 10 \text{ (Log } X)$$

where S is the shock strength and X is the total thickness of the PMMA discs in units of 1/1000 inch (mils). Note that the thinner the gap, the greater is the shock strength. A negative value means only that the gap was greater than 1000 mils.

Table 4

Minimum priming charge
Results for initiation sensitivity test

Explosive	Weight of Lead Azide (g)
PETN (E-B)	< 0.005
PETN (DuP)	< 0.005
PETN	< 0.005
HNS-II	0.05

Table 5

Minimum priming charge
Original test results for initiation sensitivity test

PETN(E-B)

mg LA	Result
40	+
30	+
20	+
10	+
5	-
5	-
5	+

End of test

PETN(DuP)

mg LA	Result
10	+
5	-
5	-
5	-
5	+

End of test

PETN

mg LA	Result
10	+
10	+
5	+
5	+
5	+
5	+
0	-
0	-
0	-
0	-
0	-
0	-

HNS-II

mg LA	Result
100	-
100	+
90	-
90	-
80	+
70	+
60	-
60	-
60	-
60	+
50	-
50	-
50	-
50	-
50	-
50	-
50	-
50	-
50	-
50	-

End of test;
take 0.05 g

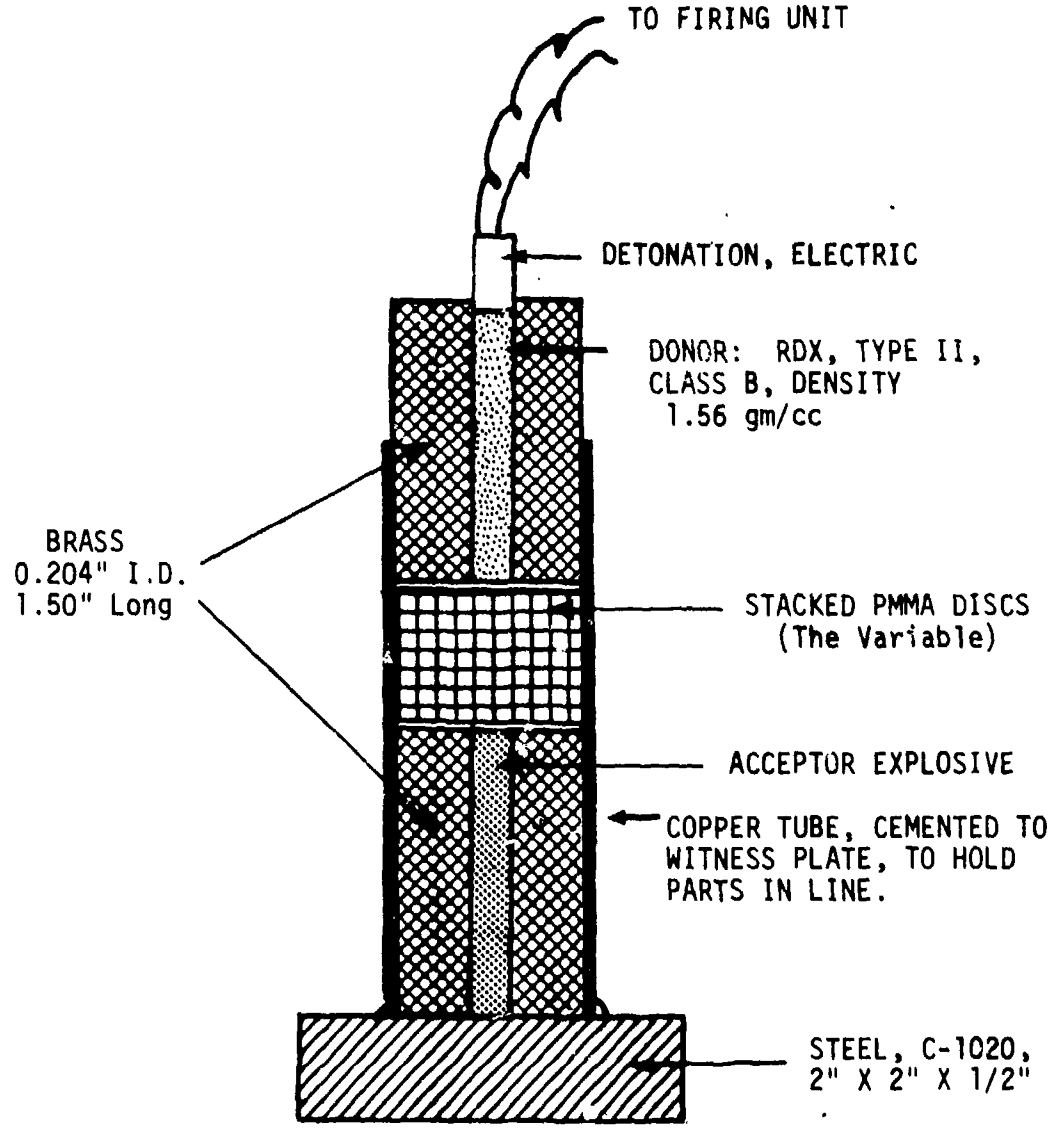

Fig 2   Small scale gap test

The Bruceton procedure was used to estimate the 50% points. For reasons of safety, regular PETN was used as reference material, rather than lead azide.

Results

The data are summarized in Table 6, together with pertinent information for the reference materials taken from Reference 4. Sensitivity to shock is a function of loading density; therefore, the values in the table are to be taken as estimates of the 50% points, for samples having the average density and standard deviations (sd) given in the second column. In the case of lead azide, the sensitivity is not markedly affected in the density range given. This range includes most densities obtained in practice. The data are also shown graphically in Figure 3.

Discussion

An evaluation of the data in Table 6 is most conveniently made visually by referring to the plots of shock strength, "S", vs % TMD (% theoretical maximum density) in Figure 3. In the plots for the PETN samples run in this work, the horizontal lines through the points indicate the standard deviations of the sample densities.

Although the "range of action" (i.e., the range of stimulus values within which one obtains both positive and negative trials, and which are used to calculate the 50% points) in this test was greater than was reported in Reference 4, the ranges were well separated from the data for the lead azide and clearly indicate the considerably lesser sensitivity to shock than is shown by azide. The values are all clustered fairly close together and near those given in Reference 4 for regular PETN, indicating that there was no gross experimental discrepancy. In light of these facts, it is concluded that both superfine PETN samples are less sensitive than lead azide to shock, and there is no marked difference between the different lots of the test explosives.

Hot Wire Sensitivity

Experimental

The test selected for this work was that for the qualification testing of primary explosives (Ref 5, 8, 9). The sample configuration is illustrated in Figure 4, and the test circuit is illustrated in Figure 5. The test variable was the voltage to which the

Table 6

Small-scale gap test results

Explosive	% Theoretical maximum density, % TMD (sd)	Sensitivity in decibangs: This Work	Sensitivity in decibangs: Reference 8
PETN (E-B)	92.7% (sd = 3.5%)	3.38	
PETN (DuP)	92.0% (sd = 2.8%)	3.63	
PETN	93.5% (sd = 3.9%)	3.63	3.17 (at 93% TMD)
Lead Azide (dextrinated)			-.3 to -.2 (for 2.775 - 3.663 g/cc)

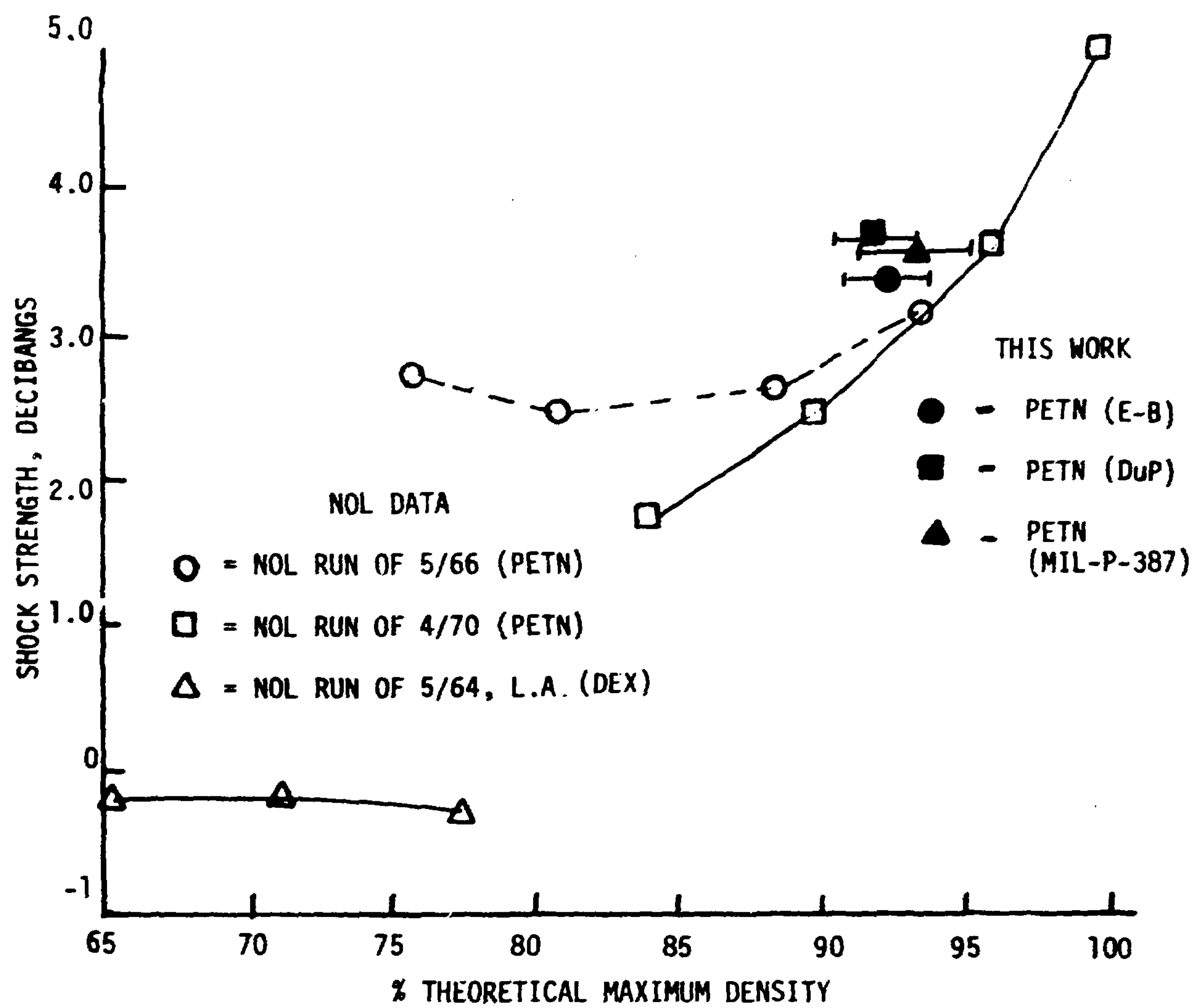

Fig 3 Shock sensitivity vs theoretical density

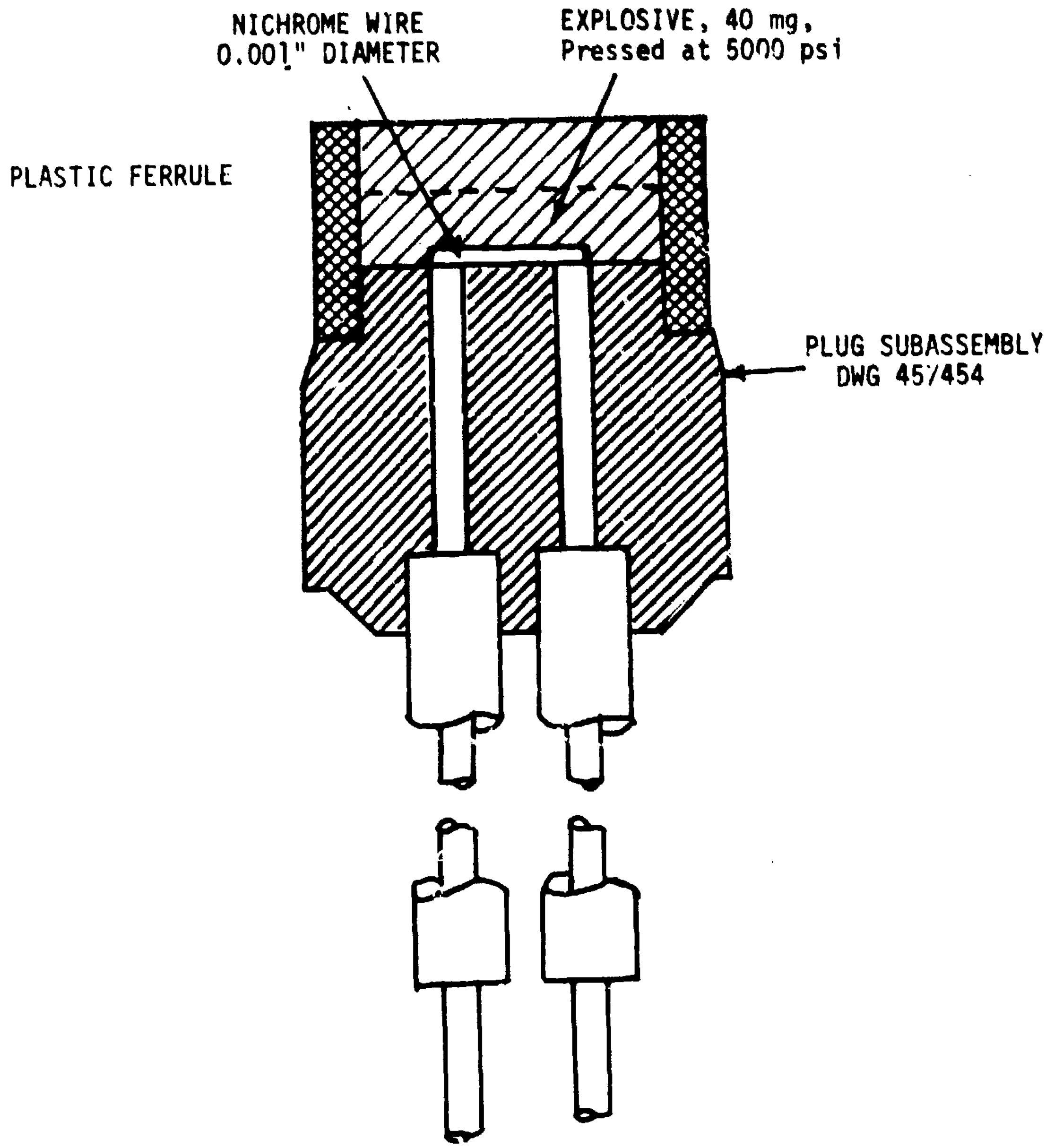

Fig 4 Hot wire ignition test

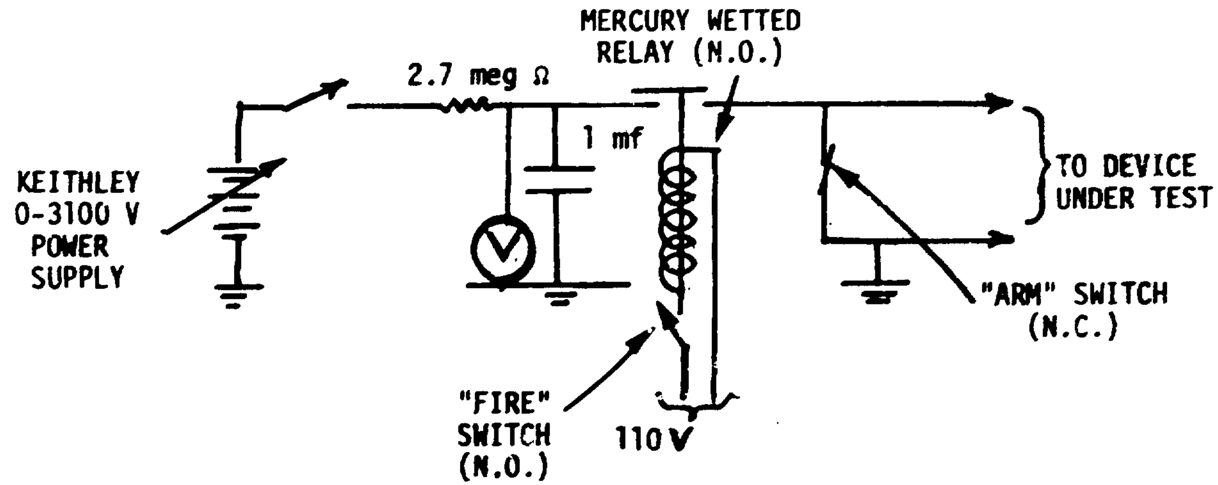

Fig 5 Schematic of hot wire sensitivity test

1.0-mF capacitor was charged, the voltage being varied in increments of 0.03 log unit, with 100 volts as the "zero" level. The Bruceton procedure was used to estimate the 50% points.

An experimental difficulty centered about the choice of the criterion for "fire" in the case of the less sensitive explosives. There was no difficulty at all with the lead azide, since a fire was very clearly evident by sound, complete consumption of the explosive sample, and shattering of the header. In the other samples, however, there were no such obvious "signals;" indeed, it was found that the wire burned out at levels not much above those required for lead azide, and at higher levels the burning out of the bare wire gave as much or more "fireworks," as was shown by the explosive samples. Accordingly, the criterion used was based on visual observation and used light emission, movement of the powder (as shown by a puff of white smoke), and sparks. There was a considerable element of subjectivity involved. In the opinion of the author, the results for the PETN and other secondary explosives are probably highly conservative, since there was no sound, or evidence of chemical decomposition products, or discoloration. The "puff of smoke" used as a partial criterion was probably dust blown out by expanding hot air.

Results

The results of the test are summarized in Table 7. Because of the ambiguity of the criterion of fire in the cases of the less sensitive explosives, the values given for these explosives should be considered as lower limits of energy for 50% probability of initiation and should not be used for quantitative comparisons with lead azide, or for comparison among themselves.

Discussion

The data indicate that all test explosives are less sensitive to short high temperature pulses than is lead azide.

Table 7

Summary of hot wire sensitivity data

Explosive	$E_{50\%}$ (Millijoules)	
Lead azide	5	"Well behaved;" no trouble
PETN (E-B)	26	In this and the following tests, the criterion of fire was "visible flame, powder movement and sound." In case of doubt, "fire" was assumed.
PETN (DuP)	21	
PETN	21	
HNS-II	30	
HMX	35	
RDX	17	

Electrostatic Sensitivity (Standard Picatinny Arsenal Test)

Experimental

The apparatus (Ref 1) consists of a sample holder and a movable electrode, a set of capacitors which can be charged by a variable voltage supply, switches, interlocks, and timing mechanisms.

The samples were 50-mg portions of loose powder. The procedure was to place the explosive in a shallow depression in a steel holder, and to allow the discharge that occurs as the needle electrode approaches the sample to arc around or through the sample. Capacitors of various sizes could be used, and these could be charged to different voltages to obtain a range of energy values as calculated by the formula:

$$E = CV^2/2 \text{ (in joules)}$$

where E is the energy, C is capacity in farads, and V is in volts.

The operator looks for any evidence of reaction, and data are recorded in terms of fraction fired out of ten samples for each energy setting. Results are expressed in terms of the minimum energy at which one or more fires are obtained in ten trials.

Results

Results are summarized in Table 8. Two groups of tests were run. In the first, an appreciable difference between the two superfine PETN samples was observed, and it was also noted that the humidity was higher than normal. It was considered advisable for these reasons to repeat the tests at a lower humidity, and to include a sample of lead azide for comparison.

Discussion

The data for the lead azide is in good agreement with published data. Presumably, therefore, the data for the other samples are valid, and indicate some difference, though not a major one, between the sensitivities of the two superfine PETN samples. The sensitivity of the PETN samples is comparable to that of HMX.

Table 8

Summary of electrostatic sensitivity data

Picatinny Arsenal Standard Test

Explosive	This Work (joules) 1st Group	2nd Group	From Reference 3
PETN (E-B)	9.76	9.76	
PETN (DuP)	1.33	4.96	
PETN		6.28 (Ref 11)	11.0
HMX	11.02		
Lead azide		0.004	0.006

Electrostatic Sensitivity (Explosives Division Parametric Test)

Experimental

The apparatus (Ref 6) is of the fixed-gap type and consists of a parallel gap assembly, a set of the capacitors and resistances, a variable source of voltage, a voltmeter, and switching devices. The gap assembly consists of a pair of smooth, parallel, steel electrodes separated by a thin sheet of dielectric material, usually 0.0075" thick electrical insulating tape in which a 3/16" diameter hole has been punched. A small amount, about 6 to 7 mg, of powder is put into the hole and the upper electrode placed over the sample. This arrangement provides good confinement and greater sensitivity than for less confined conditions.

As originally developed, capacitances approximating those likely to be found in the field--up to about 2000 pf--were usually used, and these were charged to a sufficient voltage to ensure gaseous breakdown, depending on the nature of the powder. In addition, lumped resistance, ranging from zero to several thousand ohms, may be inserted in the discharge circuit.

The circuit resistance has several effects on the discharge. Thus (depending on voltage relative to resistance) the resistance changes the discharge from an oscillatory to a unidirectional one, from arc to glow form, affects duration, and at the higher values may give rise to a relaxation oscillation instead of a single continuous gaseous discharge (Ref 6). Concurrent with these effects is a change, in a non-monotonic manner, in the fraction of stored energy that is delivered to the gap. It is to evaluate these effects on electrostatic sensitivity that this test was devised.

Various capacitors were charged to sufficient voltage to cause breakdown; however, it was necessary in these tests to use considerably larger capacitors than were usually used for primary explosives in order to obtain any positive results for PETN.

Results

Results are expressed as "zero" energies, based on twenty-five consecutive no-fires, and are summarized in Table 9.

Note the lower sensitivity of both samples of superfine PETN than lead azide at both zero circuit resistance and at high values. At the zero resistance, the discharge would be unidirectional and of glow form.

Table 9

Electrostatic sensitivities (joules) by
the parametric method

Explosive	0 Ohms	10^5 Ohms	10^6 Ohms
PETN (E-B)	0.37	1.83+	1.83+
PETN (DuP)	0.27	1.83+	1.83+
Lead azide (dextrinated)	5.6×10^{-3}		3.9×10^{-3}

Discussion

These results show a two-order-of-magnitude difference between the superfine PETN and azide at zero circuit resistance, and a three-order difference at high resistance. In this the parametric test confirms results of the Picatinny Arsenal test.

CONCLUSION AND RECOMMENDATIONS

Before summarizing, some comments about the limitations and usefulness of sensitivity data, and a cautionary note, are in order. First, current theory holds that initiation occurs when a critical volume at some local site reaches a temperature at which reaction is too fast to be quenched by the surrounding material. Thus, if energy released in such a "hot spot" can raise the temperature of the surrounding matter to reaction temperature rapidly, a runaway condition is set up. Although it is probable that the conditions controlling the transfer of energy (presumably heat) to the surrounding material are subject to considerable statistical variation, and there may be considerable question as to the validity of assumptions made in attempts to apply the theory, the underlying idea that initiation requires a localized region of high energy density does appear plausible. It is also reasonable to believe that the required critical amount of energy for the average local site is very much smaller than is very often available. It is difficult to anticipate the ways in which energy may become concentrated in a small region. The tests used are intended to simulate, so far as possible, the real hazard situation, but it should be kept in mind that such a procedure does not give a very good sampling of the situations that give rise to hazards, nor of the statistical variations in site conditions. This factor probably accounts for some of the unexplained accidents that have occurred.

A second point concerns the statistical nature of the initiation process and of sensitivity data in general. In practice we are interested in either the high or the low "tail" of the firing curve; either we want the explosive not to fire (with great certainty) or we do want it to fire (on demand) with great certainty. It is not feasible to determine such conditions directly because of the prohibitively large number of trials needed for any degree of reliability. Hence, one is constrained to work in the more accessible range, say from about 10% to 90% probability and to extrapolate in some manner to the region of interest. For this it is necessary to assume

some probability distribution function--usually the Gaussian--which implies that there is a non-zero probability at all levels despite the frequent use of "zero probability" in connection with minimum-type data. There is no assurance that the assumed function does accurately reflect initiation probability at low levels (it may be higher) and in any case there is considerable inherent uncertainty in making a long extrapolation. Experience has shown some correlation between these kinds of results and accident frequency, and in practice it is customary to compare data for a test explosive with that for materials for which there is a large body of experience. This is the practice followed in this study.

The results of these tests are summarized in Table 10, and indicate that superfine PETN is similar to standard PETN, and is less sensitive than lead azide to mechanical impact, explosive shock, short high temperature pulses, and electrostatic discharge. Nevertheless, in light of the comments above, one is definitely not justified in neglecting well established safety practices nor in being "disrespectful" in the handling of explosives, however insensitive they appear to be in tests such as these.

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Table 10

Summary of test results

Explosive	Impact: Bureau of Mines (cm)	Impact: Ball Drop (inches)	Shock: Initiation Sensitivity (mg LA)	Shock: Small-Scale Gap Test (decibangs)	Heat: Hot Wire (millijoules)	Electrostatic: Standard PA Test (joules)	Electrostatic: Expl Div Parametric (joules)
Superfine PETN (F-B)	13	45+	0.005	3.38	26	9.75/9.75	0.37 to 1.83+
PETN (DuP)	18	45+	0.005	3.63	21	1.33/4.96	0.27 to 1.83+
PETN (Mil-P-38)		45+	0.005	3.63	21	6.28	
HNS-11	15		0.05		30		
RDX					17		
HMX					35	11.02	
Lead azide	8	21.3 -			5	0.004	3.9×10^{-3} to 5.6×10^{-3}

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